National Institute of Standards & Technology

Report of Investigation

Reference Material 8418

Wheat Gluten

A Joint Material of Agriculture Canada and NIST

Distributed by the National Institute of Standards and Technology

Reference Material (RM) 8418 is intended for use in evaluating analytical methods and instruments used for the determination of major, minor, and trace constituent elements, as well as proximates, fatty acids, and calories in flour, flour products, and other similar food, agricultural, and biological materials. This material can also be used for quality assurance when assigning values to in-house control materials. RM 8418 consists of 50 g of dry powdered wheat gluten packaged in a glass bottle.

Reference Concentration Values: Reference concentration values for major, minor, and trace constituent elements are provided in Table 1. Reference concentration values for proximates, calories, and fatty acids are provided in Table 2. The reference values in Tables 1 and 2 were derived from results reported in an interlaboratory comparison exercise and by four additional collaborating laboratories, respectively. Reference values are noncertified values that are the best estimates of the true values; however, the values do not meet NIST criteria for certification and are provided with associated uncertainties that may reflect only measurement precision, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods.

Information Concentration Values: Information concentration values for additional elements, fatty acids, and total dietary fiber are provided in Tables 3 and 4. These are noncertified values with no reported uncertainties as there is insufficient information to assess uncertainties. The information values are given to provide additional characterization of the material. Use of this RM to evaluate method performance for analytes other than those with reference concentration values in Tables 1 and 2 is not warranted.

Expiration of Value Assignment: The value assignment of this RM lot is valid until **31 August 2008**, within the measurement uncertainties specified, provided the RM is handled and stored in accordance with the instructions given in this report. Value assignment is nullified if the RM is damaged, contaminated, or modified.

Maintenance of RM Value Assignment: NIST will monitor this RM over the period of its value assignment. If substantive technical changes occur that affect the value assignment before its expiration, NIST will notify the purchaser. Return of the attached registration card will facilitate notification.

Statistical support was provided by M.S. Wolynetz, Statistical Research Section, Research Program Service, Agriculture Canada and L.M. Gill of the NIST Statistical Engineering Division.

Support aspects involved with the value assignment and issuance of this RM were coordinated through the NIST Standard Reference Materials Program by J.C. Colbert and W.R. Wolf of the U.S. Department of Agriculture.

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See Report Revision History on Page 8

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RM 8418 was prepared at Agriculture Canada under the direction of M. Ihnat, Centre for Land and Biological Resources Research (CLBRR). Coordination of the technical measurements leading to the value assignment of this RM

was performed by M. Ihnat of CLBRR, Agriculture Canada, and K.E. Sharpless and S.A. Wise of the NIST Analytical Chemistry Division. Following the original analyses for elemental value assignment by the laboratories listed in Appendix A, the material was distributed by NIST to Covance Laboratories (Madison, WI), Lancaster Laboratories (Lancaster, PA), Medallion Laboratories (Minneapolis, MN), and Southern Testing and Research Laboratories (Wilson, NC) for the measurement of proximates, fatty acids, calories, and total dietary fiber.

NOTICE AND WARNING TO USERS

Storage: Until required for use, RM 8418 should be stored at room temperature in its original bottle, tightly capped, and not exposed to intense light or ultraviolet radiation.

Warning: For laboratory use only. Not for human consumption.

Instructions for Use: Prior to each use, contents of the bottle should be well mixed by gentle shaking and rolling of the bottle. A recommended minimum subsample mass of 0.5 g should be taken for elemental analysis. Moisture content should be determined on a separate subsample for conversion of analytical results to a dry-mass basis. The recommended method of drying to relate analytical results to the assigned values listed in the tables is drying for 4 h in an air oven at 85 °C. Concentrations reported in Table 1 represent total concentrations of elements in this RM. Dissolution procedures for elemental analyses should be capable of rendering a completely dissolved sample appropriate to the method and should be designed to avoid losses of elements by volatilization or by retention on decomposition and processing containers and measuring equipment. Analytical methods should be capable of measuring total levels of analytes for comparison with reference values.

PREPARATION AND ANALYSIS

Preparation: The source of material for Reference Material 8418 was food-grade-purity Whetpro-80[1] vita wheat gluten from Canadian western spring wheat flour, obtained from Ogilvie Mills Ltd., Montreal, Quebec, Canada. All preparatory work following acquisition of the commercial product was performed at the facilities of Agriculture Canada, Ottawa [1,2]. The dry bulk powder was sterilized with cobalt-60 gamma radiation to 2.0 Mrad by Atomic Energy of Canada Ltd. The material was sieved through nylon monofilament sieve cloths supported on high-density white polyethylene holders. Pairs of sieves with openings of approximately 200 µm and 50 µm were used to yield a middle cut fraction for use as the RM. This fraction was blended in a polymethylmethacrylate V-configuration blender and packaged into clean 150 mL brim capacity, colorless glass bottles with triseal (polyethylene)-lined white polypropylene screw caps. A total of 144 randomly selected units were used for physical and chemical characterization in the original analyses.

Assessment of Homogeneity: Homogeneity testing was performed on randomly selected units for 13 elements by three laboratories [2,3]. Subsamples of 0.5 g and 2.0 g were taken from a total of four units and analyzed by M. Ihnat, CLBRR, for calcium, copper, iron, potassium, magnesium, manganese, sodium, and zinc using acid digestion flame atomic absorption spectrometry [3-5]. Subsamples of 0.5 g to 6.0 g each, taken from a total of six units, were analyzed by R.W. Dabeka, Health and Welfare Canada, for cadmium, cobalt, lead, and nickel by graphite furnace atomic absorption spectrometric (GFAAS) methods following acid digestion and separation and preconcentration of the analytes using co-precipitation with ammonium pyrrolidine dithiocarbamate (all four elements) and additionally with palladium and ascorbic acid for lead [6-8]. Also, other subsamples from five units were analyzed for cadmium by GFAAS using nitric acid digests directly [9]. Fluoride was determined by the same analyst in 0.1 g subsamples from six units by an acid facilitated microdiffusion-ion specific electrode method [10]. Solid sampling GFAAS determinations were performed by M. Stoeppler and U. Bagschik, Nuclear Research Center, Jülich, Federal Republic of Germany, on a total of 40 subsamples of 0.0005 g (0.5 mg) each, from four units for copper [2,3]. In addition, the analytical results obtained from a large number of analysts (Appendix A) participating in the interlaboratory comparison exercise were assessed to provide homogeneity estimates for other elements [2,3]. No statistically significant heterogeneity was found for aluminum, barium, cadmium, calcium, chlorine, cobalt, chromium, copper, iodine, iron, lead, magnesium, manganese, mercury, molybdenum, nickel, nitrogen, phosphorus, potassium, selenium, sodium, strontium, sulfur, and zinc in sample sizes ranging from 0.1 g to 2 g, depending on the sample size typically required by the analytical technique. Data for all analytes (including the proximates and fatty acids) have been treated as though they are homogeneous, although the homogeneity of other analytes has not been investigated.

[1]Certain commercial materials and equipment are identified in order to adequately specify the experimental procedure. Such identification does not imply a recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment are the best available for the purpose.

Value Assignment: Chemical analyses to establish reference concentrations of elements were conducted in an interlaboratory comparison exercise involving Agriculture Canada and selected analysts in other laboratories (Appendix A) using analytical methods listed in Table 5. Analyses were performed by each participant on duplicate subsamples from randomly selected (typically four) units of material; subsample sizes and methods were left to the discretion of the analyst. Subsample sizes ranged from 0.001 g to 5 g, typically 0.4 g. Elemental determinations were performed on the material "as received," with conversion of results to a dry-mass basis using moisture values determined on separate 2 g subsamples by the drying procedure specified in the "Instructions for Use" section of this report.

Following the original elemental determinations, NIST distributed RM 8418 to four laboratories (Appendix B) for measurement of proximates, fatty acids, calories, and total dietary fiber. Each laboratory analyzed one portion from each of three bottles of RM 8418 using their routine methods (Table 6). Determinations were performed on the material "as received," with conversion of results to a dry-mass basis using moisture values determined on separate subsamples taken from each of the three bottles. Standard Reference Material (SRM) 1846 Infant Formula was analyzed for quality assurance.

Table 1. Reference Concentration Values of Constituent Elements

Major Constituents	Mass Fraction (%)[a]	Methods[b]
Nitrogen[c]	14.64 ± 0.21	I01, I02, J01, J02
Sulfur	0.845 ± 0.085	B02, B03, F04, J02, M02
Chlorine	0.362 ± 0.022	D01, F02, K01, K02
Phosphorus	0.219 ± 0.015	B02, B03, F01, F02, M01
Sodium	0.142 ± 0.011	A01, B01, B02, D01
Minor and Trace Constituents	**Mass Fraction (mg/kg)[a]**	**Methods[b]**
Magnesium	510 ± 47	A01, B02, B03, D01
Potassium	472 ± 61	A01, B02, B03, D01, E01
Calcium	369 ± 35	A01, B02, B03, D01, E02
Iron	54.3 ± 6.8	A01, B02, B03, D01, D03, E01, E02
Zinc	53.8 ± 3.7	A01, B02, B03, D03, E01
Manganese	14.3 ± 0.8	A01, B02, B04, D01, E01, E02
Aluminum	10.8 ± 3.0	A05, B02, B03, D01
Copper	5.94 ± 0.72	A01, A05, B02, C03, C06, E01, H01
Selenium	2.58 ± 0.19	B02, C01, C04, D01, D03, G01
Strontium	1.71 ± 0.26	B02, B03, C03, E01
Barium	1.53 ± 0.26	B02, B03, C03
Molybdenum	0.76 ± 0.09	B02, C03, C06, D01, D03, F01, H06
Nickel	0.13 ± 0.04	A16, H01
Lead	0.10 ± 0.05	A05, A16, C03, H01
Cadmium	0.064 ± 0.022	A04, A05, A16, C03, D03, H01
Iodine	0.060 ± 0.013	D03, D05, D06, F02, H03
Chromium	0.053 ± 0.013	A12, C05, D03
Cobalt	0.010 ± 0.006	A16, D01, H01
Mercury	0.0019 ± 0.0006	A10, D03

[a] Reference values are based on the dry material, dried according to instructions in this report, and are equally weighted means of results from at least two, but typically several, different analytical methods applied by analysts in different laboratories. Uncertainties are imprecision estimates expressed either as a 95 % confidence interval or occasionally (cobalt, sulfur, selenium) as an interval based on the entire range of accepted results for a single future determination, based on a sample mass of at least 0.5 g. These uncertainties, based on among-method, among-laboratory, among-unit, and within-unit estimates of variances, include measures of analytical method and laboratory imprecisions and biases and material inhomogeneity. **NOTE:** NIST has replaced the previously used term "best estimate" with "reference value."

[b] Analytical method codes and descriptions are provided in Table 5.

[c] Nitrogen results have been updated to include results from four additional laboratories (Appendix B). Each reference concentration value, expressed as a mass fraction on a dry-mass basis, is a weighted mean of the two group means from the

laboratories shown in Appendices A and B; results were weighted at 75 % and 25 %, respectively, based on the number of laboratories that provided data in the two studies. The uncertainty in the reference values is expressed as an expanded uncertainty, *U*, at the 95 % level of confidence, and is calculated according to the method described in the ISO Guide [11]. The expanded uncertainty is calculated as $U = ku_c$, where u_c is intended to represent, at the level of one standard deviation, the combined effect of between-laboratory and within-laboratory components of uncertainty. The coverage factor, *k*, is determined from the Student's *t*-distribution corresponding to the appropriate associated degrees of freedom and 95 % confidence for each analyte.

Table 2. Reference Concentration Values of Proximates, Fatty Acids (as Triglycerides), and Calories

	Mass Fraction, as received (%)[a]	Mass Fraction, dry-mass basis (%)[a]
Moisture	7.32 ± 0.94	0 (by definition)
Solids	92.68 ± 0.94	100 (by definition)
Ash	0.869 ± 0.073	0.937 ± 0.082
Protein[b]	76.7 ± 2.4	82.7 ± 3.2
Carbohydrate	10.3 ± 3.3	11.1 ± 3.4
Fat	4.8 ± 1.0	5.2 ± 1.1
Hexadecanoic Acid (C16:0) (Palmitic Acid)	0.97 ± 0.13	1.05 ± 0.14
Octadecanoic Acid (C18:0) (Stearic Acid)	0.058 ± 0.014	0.063 ± 0.014
(Z) - 9 - Octadecenoic Acid (Cl8:l) (Oleic Acid)	0.87 ± 0.24	0.94 ± 0.24
(Z,Z) - 9,12 - Octadecadienoic Acid (C18:2) (Linoleic Acid)	2.48 ± 0.72	2.68 ± 0.78
(Z,Z,Z) - 9,2,15 - Octadecatrienoic Acid (C18:3) (Linolenic Acid)	0.091 ± 0.021	0.098 ± 0.022
Eicosenoic Acid (C20:1) (Gadoleic Acid)	0.038 ± 0.011	0.041 ± 0.011
Calories[c]	(391.4 ± 7.2) kcal/100 g	(422.2 ± 5.2) kcal/100 g

[a] Each reference concentration value, expressed as a mass fraction on an as-received or dry-mass basis, is an equally weighted mean of results from the laboratories shown in Appendix B. The uncertainty in the reference values is expressed as an expanded uncertainty, *U*, at the 95 % level of confidence, and is calculated according to the method described in the ISO Guide [11]. The expanded uncertainty is calculated as $U = ku_c$, where u_c is intended to represent, at the level of one standard deviation, the combined effect of between-laboratory and within-laboratory components of uncertainty. The coverage factor, *k*, is determined from the Student's *t*-distribution corresponding to the appropriate associated degrees of freedom and 95 % confidence for each analyte. Analytical methodology information is provided in Table 6.

[b] The protein concentration was calculated from the nitrogen values reported by the laboratories shown in Appendix B using a conversion factor of 5.7; subsequent calculations of carbohydrates and calories were also based on these protein concentrations. The nitrogen values reported by the laboratories shown in Appendix B were combined with the original data for calculation of the reference value for nitrogen provided in Table 1.

[c] The value for calories is the mean of the individual caloric calculations. If the mean proximate values are used for calculation, with caloric equivalents of 9, 4, and 4 for fat, protein, and carbohydrate, respectively, the mean caloric content is 391.2 kcal/100 g and 422.0 kcal/100 g on an as-received and dry-mass basis, respectively.

Table 3. Information Concentration Values of Constituent Elements

Element	Mass Fraction (mg/kg)[a]	Methods[b]
Antimony	0.01	D01, D03
Arsenic	0.02	A07, D03
Boron	0.4	B02
Bromine	3.6	D01, E01
Fluorine	0.43	H04
Rubidium	0.4	D01, D03
Titanium	2	B02, C08, E01
Vanadium	0.04	B02, D03

[a] These analytical values, on a dry-mass basis, are estimates given strictly for information only as they are based on results of limited determinations or lack of agreement among results; no uncertainties are provided.
[b] Analytical method codes and descriptions are provided in Table 5.

Table 4. Information Concentration Values of Selected Fatty Acids (as Triglycerides) and Total Dietary Fiber

	Mass Fraction, as received (%)[a]	Mass Fraction, dry-mass basis (%)[a]
Pentadecanoic Acid (C15:0)	0.0078	0.0084
9 - Hexadecenoic Acid (C16:1) (Palmitoleic Acid)	0.0081	0.0087
Heptadecanoic Acid (C17:0) (Margaric Acid)	0.0060	0.0065
9 - Octadecenoic Acid (C18:1) (Elaidic Acid)	0.010	0.011
Eicosanoic Acid (C20:0) (Arachidic Acid)	0.0094	0.010
Docosanoic Acid (C22:0) (Behenic Acid)	0.019	0.020
13 - Docosenoic Acid (C22:1) (Erucic Acid)	0.0058	0.0062
Total Dietary Fiber	2.4	2.6

[a] These information values, reported on an as-received or dry-mass basis, are the equally weighted means of results reported by the laboratories shown in Appendix B. These values are based on results from determinations by two to four of the laboratories and are included to provide additional characterization of the material; no uncertainties are provided. Analytical methodology information is provided in Table 6.

Table 5. Analytical Methods Used by Collaborating Laboratories (Appendix A) to Determine Reference and Information Concentration Values of Elements[a]

Analytical Method	Code	Elements Determined
Acid digestion flame atomic absorption spectrometry	A01	Ca, Cu, Fe, K, Mg, Mn, Na, Zn
Acid digestion electrothermal atomic absorption spectrometry	A04	Cd
Closed vessel acid digestion electrothermal atomic absorption spectrometry	A05	Al, Cd, Cu, Pb
Acid digestion hydride generation	A07	(As)

atomic absorption spectrometry		
Closed vessel acid digestion cold vapour atomic absorption spectrometry with preconcentration	A10	Hg
Dry ashing digestion electro-thermal atomic absorption spectrometry	A12	Cr
Acid digestion coprecipitation electrothermal atomic absorption spectrometry	A16	Cd, Co, Ni, Pb
Acid digestion atomic emission spectrometry	B01	Na
Acid digestion inductively coupled plasma atomic emission spectrometry	B02	Al, (B), Ba, Ca, Cu, Fe, K, Mg, Mn, Mo, Na, P, S, Se, Sr, (Ti), (V), Zn
Closed vessel acid digestion inductively coupled plasma atomic emission spectrometry	B03	Al, Ba, Ca, Fe, K, Mg, P, S, Sr, Zn
Dry ashing inductively coupled plasma atomic emission spectrometry	B04	Mn
Acid digestion isotope dilution mass spectrometry	C01	Se
Closed vessel acid digestion isotope dilution inductively coupled plasma mass spectrometry	C03	Ba, Cd, Cu, Mo, Pb, Sr
Acid digestion dry ashing hydride generation isotope dilution inductively coupled plasma mass spectrometry	C04	Se
Dry ashing acid digestion isotope dilution mass spectrometry	C05	Cr
Acid digestion isotope dilution inductively coupled plasma mass spectrometry	C06	Cu, Mo
Acid digestion inductively coupled plasma mass spectrometry	C08	(Ti)
Instrumental neutron activation analysis	D01	Al, (Br), Ca, Cl, Co, Fe, K, Mg, Mn, Mo, Na, (Rb), (Sb), Se
Neutron activation analysis with radiochemical separation	D03	(As), Cd, Cr, Fe, Hg, I, Mo, (Sb), Se, (V), Zn
Epithermal instrumental neutron activation analysis	D05	I
Preconcentration neutron	D06	I

activation analysis		
Particle induced X-ray emission spectrometry	E01	(Br), Cu, Fe, K, Mn, (Rb), Sr (Ti), Zn
X-ray fluorescence	E02	Ca, Fe, Mn
Acid digestion light absorption spectrometry	F01	Mo, P
Dry ashing light absorption spectrometry	F02	Cl, I, P
Combustion light absorption spectrometry	F04	S
Acid digestion fluorometry	G01	Se
Closed vessel acid digestion anodic stripping voltametry	H01	Cd, Co, Cu, Ni, Pb
Acid digestion cathodic stripping voltametry	H03	I
Extraction ion selective electrode	H04	(F)
Dry ashing catalytic adsorption polarography	H06	Mo
Kjeldahl method for nitrogen -volumetry	I01	N[b]
Kjeldahl method for nitrogen- light absorption spectrometry	I02	N[b]
Combustion elemental analysis -thermal conductivity	J01	N[b]
Combustion elemental analysis with chromatographic separation -thermal conductivity	J02	N[b], S
Extraction volumetry	K01	Cl
Dry ashing volumetry	K02	Cl
Acid digestion gravimetry	M01	P
Dry ashing gravimetry	M02	S

[a] Letter codes refer to classes of similar methods; number codes refer to specific variants. Elements in parentheses have only information values in this RM. **NOTE:** NIST has replaced the previously used term "best estimate" with "reference value."

[b] See Table 6 for additional information.

Table 6. Methods Used by Collaborating Laboratories (Appendix B) for the Determination of Proximates, Fatty Acids, Calories, and Total Dietary Fiber

Ash	mass loss after ignition in a muffle furnace
Calories	calculated; [(9 × fat)+ (4 × protein) + (4 × carbohydrate)]
Carbohydrate	calculated; [solids – (protein + fat + ash)]
Fat	sum of individual fatty acids
Fatty acids	hydrolysis followed by gas chromatography
Moisture	mass loss after drying in a vacuum oven (2 laboratories); mass loss after drying in a forced-air oven (2 laboratories)
Nitrogen	Dumas (1 laboratory); modified Dumas (1 laboratory); Kjeldahl (2 laboratories). Note that in the original elemental determinations, laboratories provided results using Kjeldahl, combustion - thermal conductivity, and combustion - chromatographic separation - thermal conductivity.
Protein	calculated from nitrogen using a factor of 5.7
Solids	calculated; (sample mass – moisture)
Total dietary fiber	enzymatic digestion followed by gravimetry

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[11] *Guide to the Expression of Uncertainty in Measurement,* ISBN 92-67-10188-9, 1st Ed., ISO, Geneva, Switzerland, (1993); see also Taylor, B.N. and Kuyatt, C.E., "Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results," NIST Technical Note 1297, U.S. Government Printing Office, Washington DC, (1994); (available at http://physics.nist.gov/Pubs/).

Report Revision History: 04 October 99 (This technical revision reports the addition of reference and information values for calories, fatty acids, and total dietary fiber.); 18 February 94 (editorial revision); 24 September 93 (original report date).

Users of this RM should ensure that the report in their possession is current. This can be accomplished by contacting the SRM Program at: Telephone (301) 975-6776 (select "Certificates"), Fax (301) 926-4751, e-mail srminfo@nist.gov, or via the Internet http://ts.nist.gov/srm.

Appendix A. Collaborating Analysts for Elemental Determinations

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Appendix B. Collaborating Laboratories for Proximate, Fatty Acid, Total Dietary Fiber, and Calorie Determinations

Covance Laboratories, Madison, WI, USA.

Lancaster Laboratories, Lancaster, PA, USA.

Medallion Laboratories, Minneapolis, MN, USA.

Southern Testing and Research Laboratories, Wilson, NC, USA.